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16. ABSTRACT

In 1985, the Quality Assurance Division conducted the National Audits for Stationary Source Test Methods. The audit materials consisted of a disposable gas cylinder for Method 3 (Orsat analyzer), a calibrated orifice for Method 5 (DGM only), five simulated liquid samples each for Method 6 (SO_2) and Method 7 (NO_x), and two coal samples for Method 19A. Participating laboratories sent their data to the Source Branch and in return received a written report comparing their results to EPA's.

In the Method 3 audit, each parameter had only one concentration. The mean for CO_2 was 4.0% from the expected (EPA) value. The mean for O_2 was 0.4%.

In the Method 5 audit, the mean value for all participants differed by 10.6% from the expected value. For the Method 6 audit, the average mean differed by 10% from the expected value, and in the Method 7 audit the average mean differed by 4.0%.

In the two coal audits, the parameters measured were sulfur, moisture, ash, and Btu content. On the average for the sulfur analysis, 87% of the participants measured within 10% of the expected value; for Btu, 98% of the participants measured within 10% of the expected value.

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A SUMMARY OF THE 1985 EPA NATIONAL PERFORMANCE AUDIT PROGRAM ON SOURCE MEASUREMENTS

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FOREWORD

Measurement and monitoring research efforts are designed to anticipate potential environmental problems, to support regulatory actions by developing an in-depth understanding of the nature and processes that impact health and the environment, to provide innovative means of monitoring compliance with regulations, and to evaluate the effectiveness of health and environmental protection regulations through the monitoring of long-term trends. The Environmental Monitoring Systems Laboratory, Research Triangle Park, North Carolina, has responsibility for: assessment of environmental monitoring technology and systems; implementation of Agency-wide quality assurance programs for air pollution measurement systems; and supplying technical support to other groups in the Environmental Protection Agency, including the Office of Air and Radiation and the Office of Toxic Substances.

The major task of this study was to report the results of the national quality assurance audit program for stationary source test methods. Audits were designed to estimate the minimal analytical and computational accuracy that can be expected with Method 3 (carbon dioxide and oxygen), EPA Method 5 (dry gas meter only), Method 6 (sulfur dioxide), Method 7 (nitrogen oxides), and Method 19 (coal). Statistical analysis was used to characterize the data.

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ABSTRACT

In 1985, the Quality Assurance Division conducted the National Audits for Stationary Source Test Methods. The audit materials consisted of: a disposable gas cylinder for Method 3 (Orsat analyzer), a calibrated orifice for Method 5 (dry gas meter only), five simulated liquid samples each for Method 6 (SO_2) and Method 7 (NO_x), and two coal samples for Method 19A. Participating laboratories sent their data to the Source Branch and in return received a written report comparing their results to EPA's.

In the Method 3 audit, each gas component had only one concentration. The mean CO_2 value for all participants differed by 4.0 percent from the expected (EPA) value, and the mean for O_2 was 0.4 percent from the expected value.

In the Method 5 audit, the mean value for all participants differed by 10.6 percent from the expected value. For the Method 6 audit, the average mean differed by 10 percent from the expected value. The average mean in the Method 7 audit was 4 percent from the expected value.

In the two coal audits, the parameters measured were sulfur, moisture, ash, and Btu content. On the average for the sulfur analysis, 87 percent of the participants measured within 10 percent of the expected value; for Btu, 98 percent of the participants measured within 10 percent of the expected value.

This report includes the results of the performance audits done during the period from January to December 1985.

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We express our appreciation to the laboratories that participated in the National Performance Audit Program for Stationary Sources. Thanks also to the staff of the Standards Laboratory/Performance Evaluation Branch/ Environmental Monitoring Systems Laboratory (EMSL), who did the acceptance testing of the audit samples, and to the programmers of the Monitoring and Assessment Division/EMSL for providing the data systems to store and evaluate the data.

SECTION 1

INTRODUCTION

The Environmental Protection Agency's (EPA's) Environmental Monitoring Systems Laboratory (EMSL) at Research Triangle Park, North Carolina, established an audit program in 1977 to evaluate the performance of companies that conduct compliance testing using EPA reference methods. The audits check the participants' analytical accuracy in applying the analytical phase of EPA Reference Methods 3, 6, 7, and 19A and the calibration accuracy of the Method 5 control console.[1] Accuracy is defined as the percent difference between a participant's analytical results and the EPA expected value. By participating in this free and voluntary program, testing companies can compare their performance to other laboratories conducting similar measurements.

Source Test Methods 3, 5, 6, and 7 were each audited once and Method 19A twice in 1985. Each participating laboratory received an audit package consisting of the audit sample, a data card, instructions, and an envelope for returning the data to EPA. A label for returning the audit device was included with the Method 5 audit package. Participants had 8 weeks to use the audit material and return their data to EPA. At the end of this period, all data received were statistically analyzed to determine the accuracy with respect to the EPA expected value and those obtained by the participants (see Appendix A).

The Quality Assurance Division of EMSL also maintains a limited repository of samples for the EPA Methods 3, 6, 7, and 19A that are available to source testing laboratories for such purposes as training new personnel or conducting quality control checks. Because the expected values for these samples are included with the analysis instructions, there is no requirement for the data to be returned to EPA. We recommend that source testing laboratories use this sample repository to help improve their overall analytical performance.

This report summarizes the results obtained in the 1985 source audits.

SECTION 2

SUMMARY

In 1985, EPA's EMSL at Research Triangle Park, North Carolina, conducted National Quality Assurance Audits for Stationary Source Test Methods 3 (Orsat analyzer), 5 (dry gas meter only), 6 (SO_2), 7 (NO_x), and 19A (coal). Industrial laboratories, contractors, foreign laboratories, as well as local, State, and Federal agencies participated.

The results of the 1985 audit of Method 3 are summarized in Table 1. Participants analyzed the gas sample twice for percentages of carbon dioxide (CO_2), oxygen (O_2), and carbon monoxide (CO). The mean values of CO_2 and O_2 differed by 4 percent and 0.4 percent from the expected values, respectively. In contrast, the mean values for CO differed as much as 63 percent from the expected values.

TABLE 1. PARTICIPANTS' RESULTS FROM METHOD 3 AUDIT (ALL DATA--NO OUTLIERS REMOVED)

Type of sample	Audit date	Parameter	No. of analyses	Replicate	EPA true value	Participant results		
						Mean	Median	Std.dev.
Small cylinder (gas)	0785	% CO_2	46	1	7.00	6.76	6.60	0.97
			45	2	7.00	6.68	6.60	0.88
		% O_2	47	1	12.00	11.98	12.00	1.38
			46	2	12.00	12.07	12.00	1.33
		% CO	37	1	0.60	0.96	0.50	2.92
			36	2	0.60	1.00	0.50	2.97

One audit of Method 5 was conducted in 1985. The overall results (no outliers removed) are summarized in Table 2. The mean for all participants was 10.6 percent from the expected value and the standard deviation was 102.1 percent. After the removal of 7 percent of the data statistical outliers, the mean was 2.3 percent from the expected values and the standard deviation was 1.7 percent. The participants' performance based on the standard deviation of all data was poor; however, when the 7 percent of the outliers was removed, the performance was consistent with previous years.

TABLE 2. METHOD 5 AUDIT 0685 - SUMMARY STATISTICS

	n	Mean %	Median %	Std. dev. %
All data	633	10.6	2.2	102.1
Outliers removed	588	2.3	2.0	1.7

Table 3 represents the data (no outliers removed) from the Methods 6 and 7 audits. In the Method 6 audit, the procedure requires the participants to determine the sulfate content in five aqueous solutions using the titration procedure. For four out of five concentrations, the mean of the participants was 10 percent higher than the expected value. In contrast, the median differed by 2 percent. In the Method 6 audit, 60 to 85 percent of the participants achieved an accuracy within 5 percent of the expected value.

The Method 7 audit procedure requires that the participants determine the nitrate content in five aqueous solutions. For each concentration, the mean of the participants was less than 4 percent from the expected value, and the median differed by less than 2 percent. In this audit, 70 percent of the participants achieved an accuracy within 10 percent of the expected value for all five concentrations.

TABLE 3. PARTICIPANTS' RESULTS FROM METHODS 6 AND 7 AUDITS
(ALL DATA--NO OUTLIERS REMOVED)

Type of sample	Audit date	No.of analyses	EPA true value (mg/DSCM)	Participant results		
				Mean	Median	Std. dev.
Aqueous sulfate	0585	89	198.3	243.8	201.5	263.8
		91	221.2	245.5	225.0	104.5
		90	701.6	723.7	698.3	247.9
		91	1250.7	1332.6	1240.1	547.5
		89	1326.9	1426.1	1323.1	595.3
Aqueous nitrate	0485	72	99.5	97.9	101.1	24.4
		73	119.5	118.3	119.0	23.0
		73	298.6	293.8	298.0	49.5
		72	338.4	344.8	339.4	73.8
		73	537.5	518.4	534.0	112.9

Table 4 represents the data from Methods 6 and 7 after the removal of outliers. Two percent of the Method 6 data and 3 percent of the Method 7 data were classified as outliers. It is important to note that even with the statistical outliers removed, the performance of this year's Method 6 data (based on the standard deviation) is not as good as in previous years.

TABLE 4. PARTICIPANTS' RESULTS FROM METHOD 6 AND 7 AUDITS (OUTLIERS REMOVED)

Type of Sample	Audit Date	No. of Analyses	EPA true value (mg/DSCM)	Participant results		
				Mean	Median	Std. dev.
Aqueous sulfate	0585	88	198.3	217.6	201.3	91.9
		88	221.2	227.6	224.6	25.2
		89	701.6	699.6	697.8	95.9
		89	1250.7	1252.5	1240.0	103.6
		87	1326.9	1338.1	1322.2	117.0
Aqueous nitrate	0485	69	99.5	101.5	101.9	17.5
		71	119.5	120.8	119.1	17.7
		71	298.6	299.4	298.5	36.3
		70	338.4	342.9	339.4	49.5
		71	537.5	530.9	534.0	84.8

Table 5 summarizes the results of the two coal audits that were conducted in 1985. Participants analyzed each coal sample in duplicate for percentages of sulfur, moisture, and ash, and for gross calorific value (Btu/lb). The means of the ash, moisture, and sulfur content were within 6 percent of the expected value except for a 20 percent difference on the lowest sulfur concentration. An accuracy of 1 percent was achieved on the Btu content. The Btu content as measured by the participants is lower than the EPA value because coal oxidation is not prevented in the audit sample. The Btu content is a good example of the accuracy to which a measurement can be made when that measurement receives good laboratory quality control.

TABLE 5. PARTICIPANTS' RESULTS FROM METHOD 19A COAL AUDITS
(ALL DATA--NO OUTLIERS REMOVED)

Type of sample	Audit date	Parameter	No.of analyses	EPA value	Participant results		
					Mean	Median	Std.dev.
Coal	0385	% S	111	1.62	1.60	1.59	0.14
	0985		108	0.72	0.86	0.70	1.56
	0385		110	4.97	4.75	4.76	0.35
	0985		106	2.35	2.29	2.29	0.23
	0385	% H_2O	111	3.86	3.60	3.70	0.52
	0985		106	1.61	1.75	1.53	1.86
	0385		112	3.93	3.97	3.95	1.65
	0985		108	17.45	16.73	17.41	2.61
	0385	% Ash	111	11.36	11.37	11.33	0.45
	0985		107	4.33	4.25	4.37	0.44
	0385		110	20.32	19.92	20.07	1.52
	0985		105	17.92	17.59	17.83	2.34
	0385	Btu/lb	108	11,414.0	11,379.7	11,383.5	256.61
	0985		102	12,300.0	12,252.9	12,273.0	183.77
	0385		109	13,054.0	13,011.1	13,025.0	261.53
	0985		104	12,374.0	12,179.3	12,319.0	573.78

SECTION 3

METHOD 3 AUDIT

The Method 3 audit checks participants' ability to analyze a gas sample using an Orsat analyzer. The audit package consists of a disposable cylinder that contains a 4-liter (L) sample of CO_2, O_2, and CO. The analyst expels the gas into the Orsat analyzer using the positive pressure of the cylinder. The gas sample is quantitatively analyzed for percentage of CO_2, O_2, and CO.

In the 1985 audit, 44 percent of the 103 laboratories receiving the audit package returned data. Table 6 shows the total number of laboratories requesting participation and the number that returned data for the Method 3 audit.

TABLE 6. METHOD 3 AUDIT PARTICIPANTS

Category	No. receiving samples	No. returning data
Contractors	57	16
Industry	28	15
Foreign	1	1
Federal	2	2
State	12	8
Local	3	3
Total	103	45

Table 7 summarizes the Method 3 audit results. Each laboratory was asked to analyze the sample in duplicate. Five and ten percent accuracy were chosen for the reporting criteria for each of the parameters. Each parameter had only one concentration.

In the 1985 audit, 35 percent of the reporting laboratories achieved an accuracy within 5 percent for the CO_2, which was a decrease from the last audit.[4] Ninety percent of the laboratories achieved an accuracy within

5 percent of the expected value for the O_2 analysis. For the CO analysis, only 33 percent of the laboratories achieved an accuracy within 5 percent, and 21 percent of the laboratories did not report a value for CO.

TABLE 7. SOURCE METHOD 3 AUDIT--0785

Expected value	No. of analyses*	Laboratories accurate within 5% (%)	Laboratories accurate within 10% (%)
7.00	(1) 46	35	85
	(2) 45	30	89
12.00	(1) 47	89	92
	(2) 46	91	94
0.60	(1) 37	32	32
	(2) 36	36	36

* Numbers in parentheses indicate first and second analyses.

SECTION 4

METHOD 5 DRY GAS METER AUDIT

In the Method 5 audit procedure, participants use a calibrated orifice to check the calibration of the dry gas meter in their EPA Method 5 control console (meter box). They insert the orifice in the Method 5 meter box, allow the box to warm up, and then make three 15-min volume measurements. The participants convert each of the three volumes to cubic meters at standard conditions using the formula specified in Eq.# 5.1 of Method 5 (Appendix A, 40 CFR 60) and record them on the data card. Then they return the orifice and the data card to EPA, where the data undergoes statistical analysis.

In the 1985 audit, 59 percent of the 160 laboratories that received the audit package returned data. Table 8 shows the categories of participants, the number of participants who requested participation in the Method 5 audit, and the number who actually returned data.

TABLE 8. METHOD 5 AUDIT PARTICIPANTS

Category	No. receiving samples	No. returning data
Contractors	81	37
Industry	51	35
Foreign	4	2
Federal	2	2
State	18	14
Local	4	4
Total	160	94

Figure 1, a cumulative histogram, shows the accuracy obtained by participants in the Method 5 audit, expressed as the percent difference from the expected (EPA) value at various levels of accuracy. The Code of Federal Regulations[1] requires that the dry gas meter be calibrated within an accuracy of 2 percent. Figure 2 shows that 47 percent of the reporting laboratories attained this accuracy. These results are not as good as those from 1984. Table 9 is a summary of previous audits that, like Figure 2, shows a decline from 1984.

TABLE 9. PREVIOUS RESULTS OF METHOD 5 AUDIT

Audit	Number of analyses	Mean (% from EPA value)	Median	Std. dev.
0382	827	7.6	2.5	39.5
0982	769	12.5	2.5	81.4
0383	763	5.7	2.2	32.7
0983	614	4.1	1.9	21.3
0684	631	3.1	1.9	4.9
0685	633	10.6	2.2	102.1

The histogram in Figure 3 depicts the individual results from all participants of the 1985 audit with the mean and median values. The majority of the laboratories reported values lower than the EPA value.

The standard deviation of the triplicate analysis (repeatability) by each laboratory indicated that 71 percent of the standard deviations for each set were within 0.3 percent. Four percent of the 1985 data were identified as outliers using Chauvenet's Criterion.[5] Before the outliers were removed, the mean value (absolute) differed by 10.6 percent from the expected value. After deletion of outliers, this value was reduced to 2.3 percent.

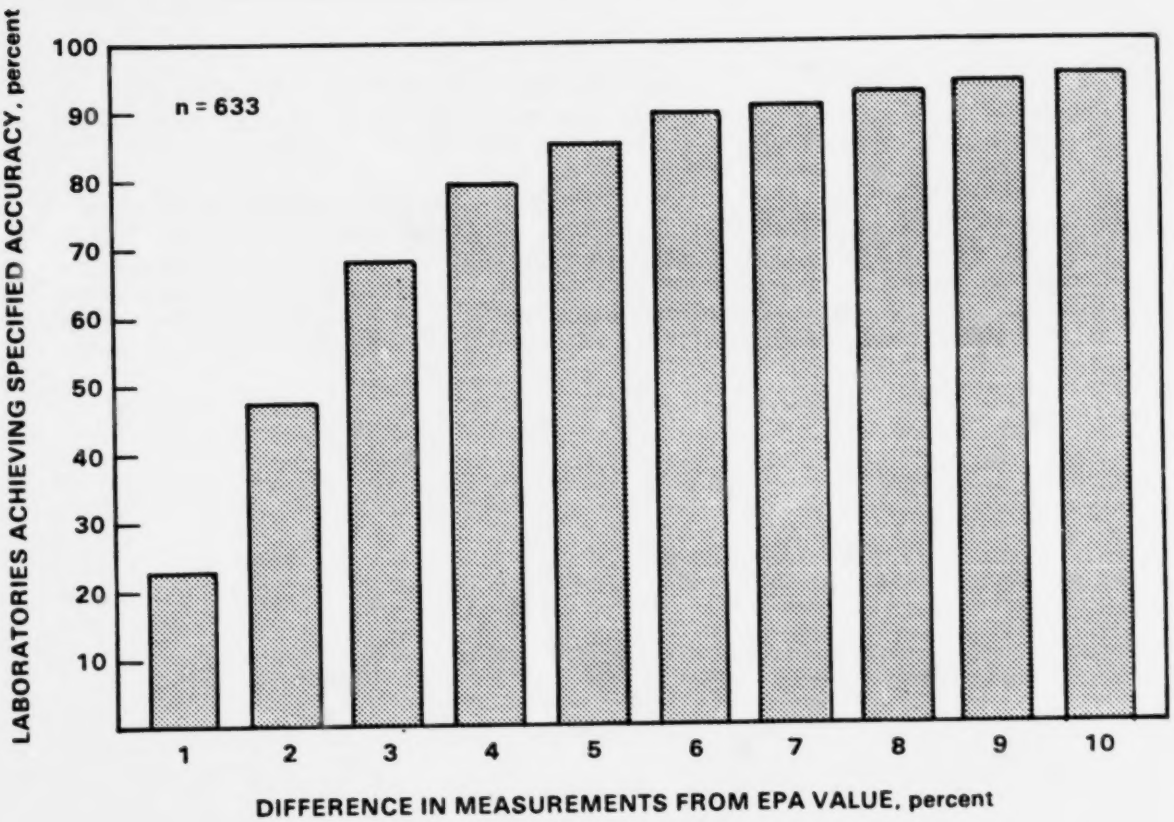

Figure 1. Cumulative accuracy for participants in method 5 audit, 0685.

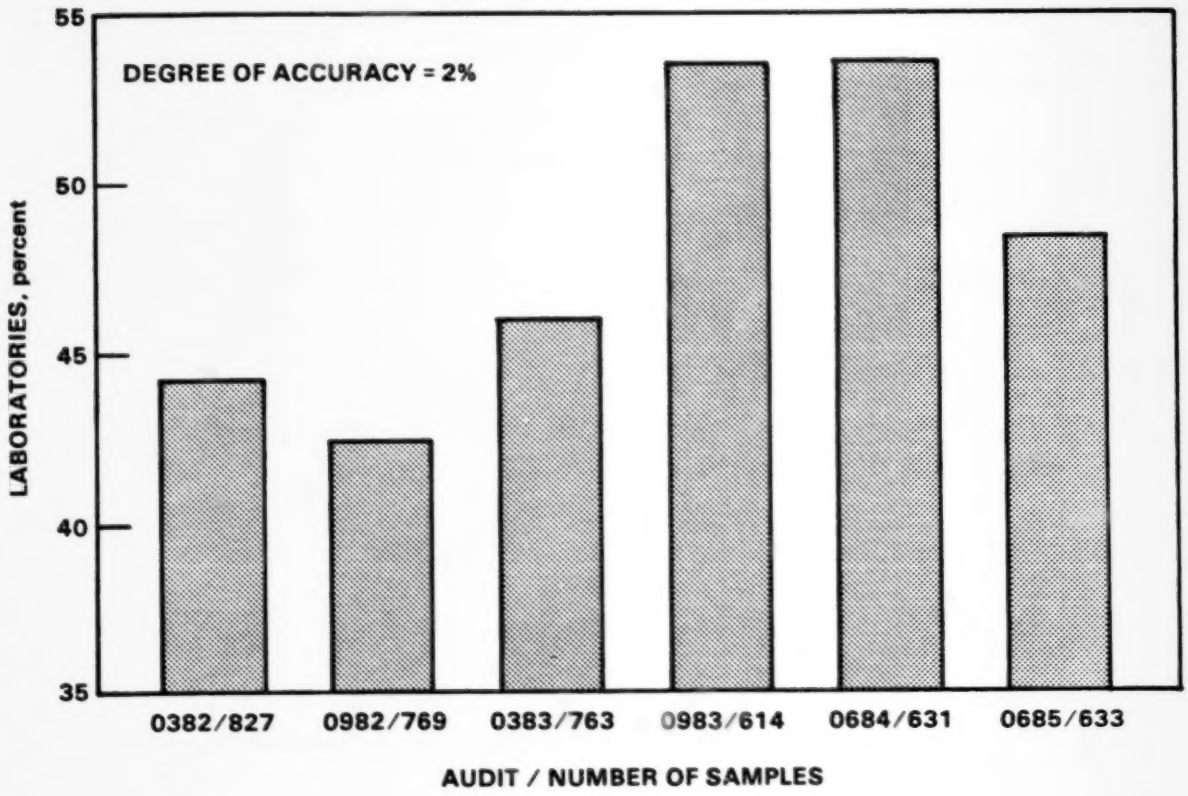

Figure 2. Previous results of Method 5 audit.

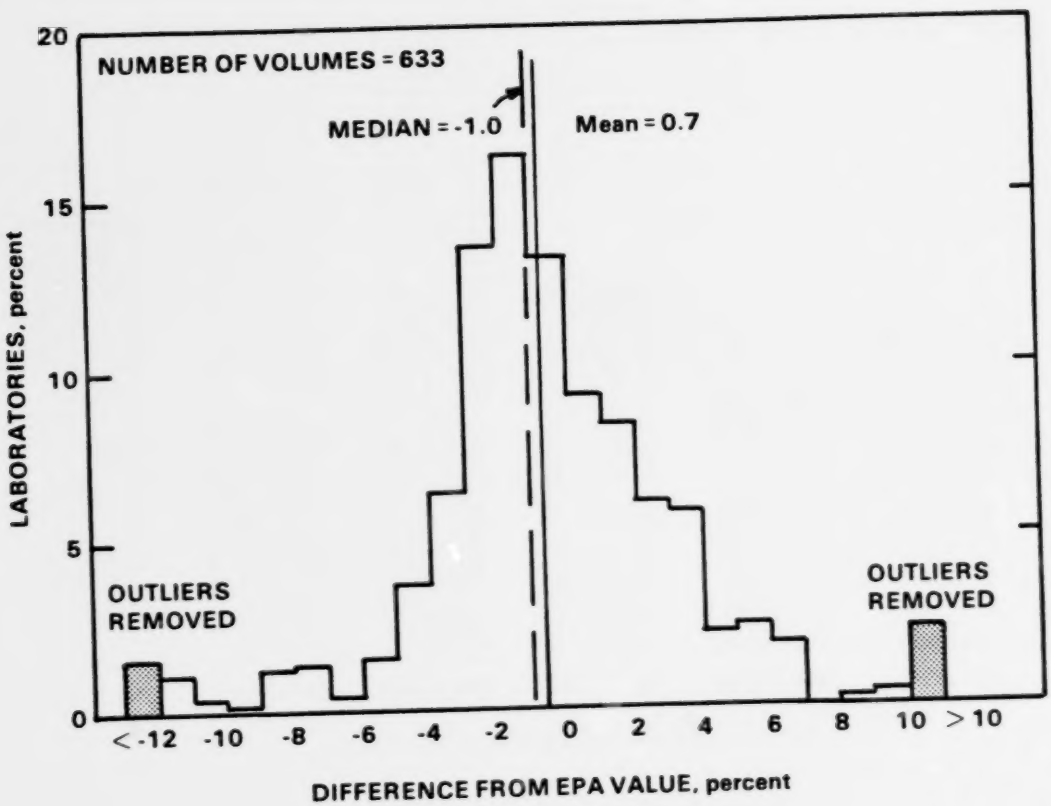

Figure 3. Results of method 5 audit, 0685.

SECTION 5

METHOD 6 AUDIT

The Method 6 audit checks participants' ability to quantitatively analyze Method 6 samples for sulfur content. The audit set consists of five aqueous dilutions of 10 N sulfuric acid in 25 milliliters (mL) sealed glass ampoules. The analyst withdraws 5 mL from each ampoule, adds 30 mL of 3 percent hydrogen peroxide, and dilutes this sample to 100 mL with distilled water. A 20-mL aliquot is then withdrawn from the diluted sample, 80 mL of 100 percent isopropanol and thorin indicator are added, and the sample is titrated with barium perchlorate to a pink endpoint. In calculating the results, the participants assume an original sample volume of 100 mL and a sample volume of 0.021 dry standard cubic meter of stack gas.

Table 10 shows the categories of the participants and compares the total number of participants requesting participation with the number returning data. In the 1985 audit, 66 percent of the 137 laboratories that received the audit package returned data.

TABLE 10. METHOD 6 AUDIT PARTICIPANTS

Category	No. receiving samples	No. returning data
Contractors	74	42
Industry	40	30
Foreign	3	2
Federal	1	1
State	13	11
Local	6	5
Total	137	91

Table 11 shows the percentage of laboratories that achieved 2 percent and 5 percent accuracy for each of the five different concentrations in the Method 6 audit. At least 62 and 67 percent of the reporting laboratories achieved an accuracy within 5 percent for the two lower concentrations, but 83 percent of the laboratories showed an improvement on the higher concentrations. Five percent is used as the criterion because it was established as the criterion for source SO_2 compliance audit samples.

TABLE 11. SUMMARY OF SOURCE SO_2 AUDITS--0585

Concentration (mg/DSCM)	± 2% (%)	± 5% (%)
198.3	28.1	61.8
221.2	34.1	67.0
701.6	58.9	83.3
1250.7	39.6	87.9
1326.9	64.0	86.5
N	91	

Figure 4 shows the mean values for each SO_2 concentration and the confidence intervals (CI). Each CI is calculated using the 95 percent confidence level. The CI for the lowest concentration is ± 22.5 percent; whereas the CI for the highest concentration is ± 8.7 percent. Overall, the CI's for the SO_2 were higher than expected for this method because of the high standard deviations.

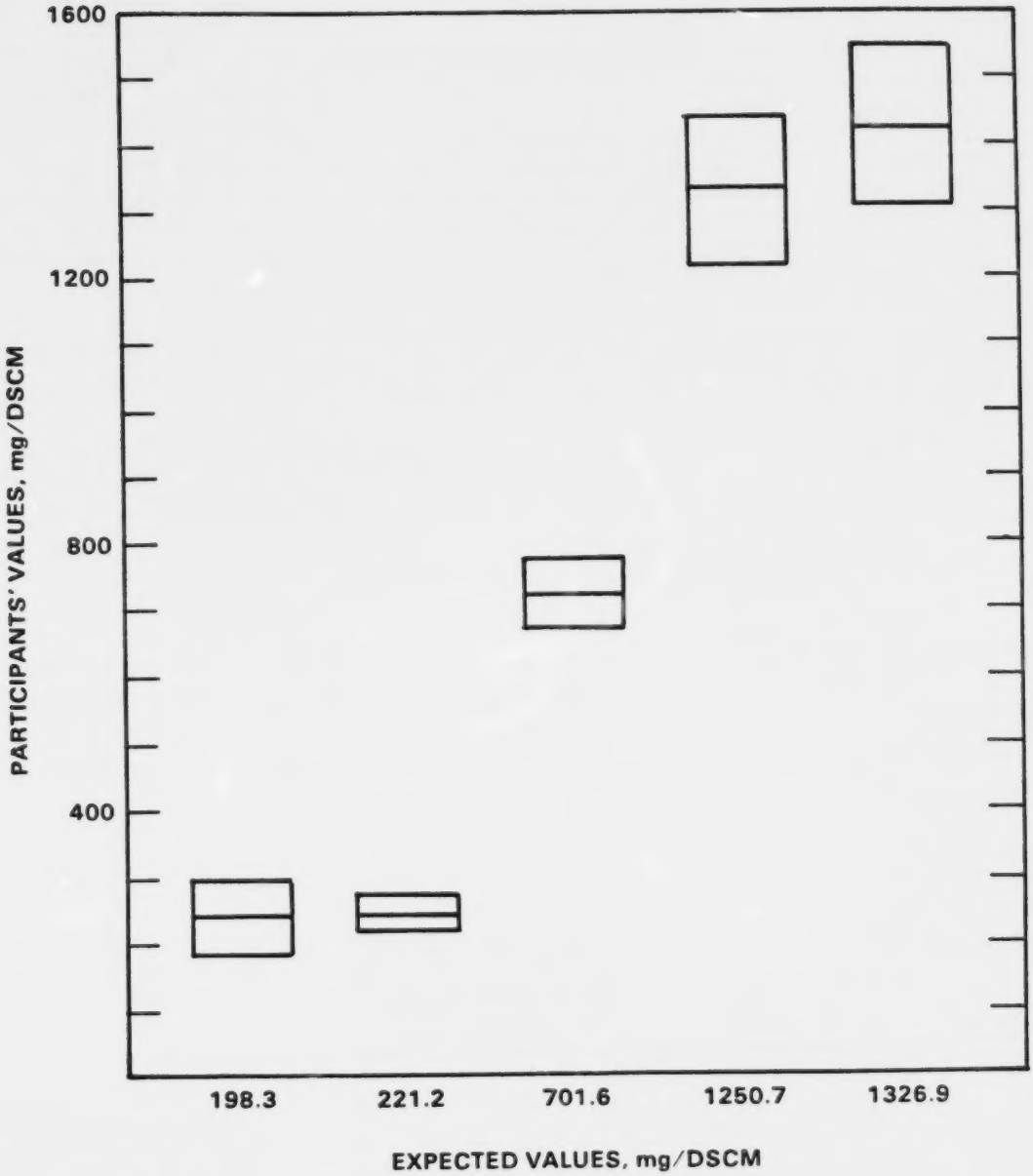

Figure 4. SO_2 expected values compared to participants' values.

SECTION 6

METHOD 7 AUDIT

The Method 7 audit checks participants' ability to quantitatively analyze Method 7 samples for nitrate content. The NO_x audit set consists of five aqueous dilutions of a potassium nitrate solution in 25-mL glass ampoules that are autoclaved after sealing so that bacteria that might attack the nitrate are destroyed. The analyst withdraws 5 mL of solution from an ampoule, adds this with 25 mL of Method 7 absorbing solution to a flask, adjusts the pH with sodium hydroxide, and dilutes to 50 mL with distilled water. A 25-mL aliquot is withdrawn from the diluted sample, placed in an evaporating dish, and analyzed as described in Section 4.3 of Method 7.[1] After this treatment is completed, the absorbance is measured at 410 nanometers (nm) with a calibrated spectrophotometer. In calculating the concentrations present, the participant assumes that 2000 mL of stack gas was sampled.

Table 12 shows the total number of laboratories requesting participation and the number that returned data for the 1985 Method 7 audit. Sixty-two percent of the 117 laboratories receiving the audit package returned data.

TABLE 12. METHOD 7 AUDIT PARTICIPANTS

Category	No. receiving samples	No. returning data
Contractors	67	36
Industry	36	25
Foreign	2	2
Federal	1	0
State	6	6
Local	5	4
Total	117	73

The percentage of laboratories that achieved 5 and 10 percent accuracy for each of the five concentrations is shown in Table 13. Ten percent is used as the criterion because it was established as the criterion for the source NO_x compliance audit samples. Sixty percent of the reporting laboratories achieved an accuracy within 10 percent on the lowest concentration, and 70 to 80 percent achieved an accuracy within 10 percent on the highest concentrations.

TABLE 13. SUMMARY OF SOURCE NO_x AUDIT--0485

Concentration (mg/DSCM)	± 5% (%)	± 10% (%)
99.5	41.7	58.3
119.5	49.3	69.9
298.6	54.8	80.8
338.4	51.4	72.2
537.5	53.4	71.2
N	73	

Figure 5 shows the means for each NO_x concentration and the CI's. The CI's are calculated the same as for SO_2. The CI for the lowest concentration is ± 5.8 percent, whereas the CI for the highest is 5.0 percent. Overall, the CI's for the NO_x were very low because of low standard deviations.

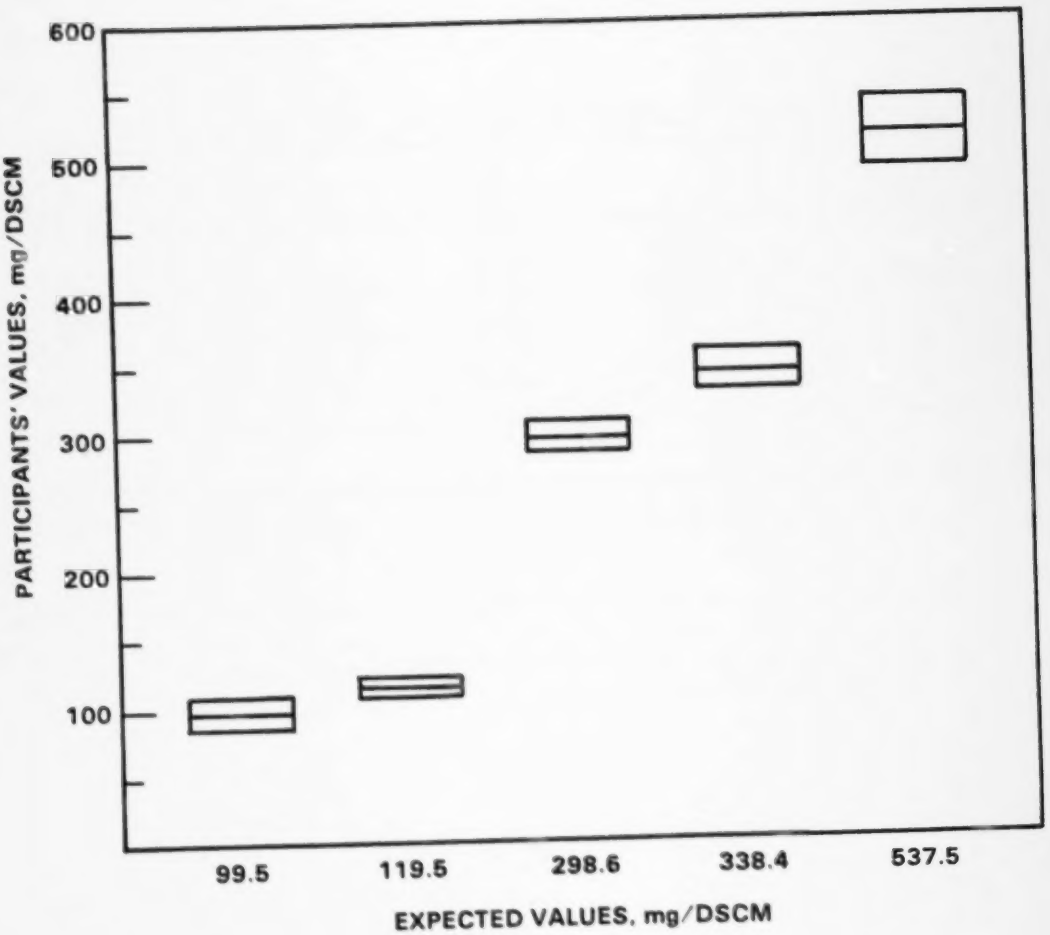

Figure 5. NO_x expected values compared to participants' values.

SECTION 7

METHOD 19A COAL AUDIT

Standards of performance for newer electric utility steam generators (Subpart Da of 40 CFR 60) allow coal sampling and analysis to serve as an acceptable method for determining scrubber inlet flue gas sulfur concentration. The coal audit checks participants' ability to analyze coal samples for sulfur, ash, moisture, and Btu content.

The coal audit samples consisted of two samples each with 50 grams (g) of 60-mesh coal but with different parameter levels. The following American Society for Testing and Materials (ASTM) procedures[6] were recommended, but not required, for participants' use in analyzing the coal samples:

- ASTM D-3177 (Standard Test Method for Total Sulfur in the Analysis of Coal and Coke)
- ASTM D-3174 (Standard Test Method for Ash in the Analysis Sample of Coal and Coke)
- ASTM D-3173 (Test for Moisture in the Analysis Sample of Coal)
- ASTM D-2015 (Standard Test Method for Gross Calorific Value of Solid Fuel by the Adiabatic Bomb Method).

Participants measured the parameters and reported their results for moisture (%) on an as-received basis, and their results for sulfur (%), ash (%), and gross calorific value (Btu/lb) on a dry basis.

In both audits, 80 percent of the laboratories that received the audit package returned data. Seventy-three of the same laboratories participated in both audits and returned data. Table 14 shows the total number of laboratories requesting participation and the number that returned data for coal audits 0385 and 0985.

TABLE 14. COAL AUDIT PARTICIPANTS

Category	No. requesting samples		No. returning data	
	0385	0985	0385	0985
Contractors	57	52	42	42
Industry	42	37	37	33
Federal	1	1	0	0
State	11	12	10	10
Local	4	5	3	5
Total	115	107	92	90

Tables 15 and 16 summarize the coal audit results. The number of analyses is greater than the number of participants because some companies had more than one laboratory participating. In this case, each laboratory received its own set of samples and was asked to analyze the samples in duplicate. Accuracies of 5 and 10 percent were chosen as the reporting criteria for each of the four parameters (sulfur, moisture, ash, and gross calorific content).

In the 0385, 90 percent of the laboratories were able to analyze both sulfur samples within 10 percent of the expected value. In the 0985 audit, 76 percent of the laboratories were able to analyze the sulfur content of the lower concentration sample within 10 percent of the expected value; however, 90 percent were able to analyze the high level sample within 10 percent of the expected value. Seventy percent were within the 10 percent criterion for both moisture concentrations in the 0385 audit, whereas 89 percent in the 0985 audit achieved 10 percent for the higher moisture concentration, but only 55 percent of the laboratories achieved 10 percent on the lower concentration. For the ash analysis and Btu content, 87 to 100 percent of the reporting laboratories were able to achieve an accuracy within 10 percent for both sample concentrations.

Comparing the 0385 audit to the 0985 audit, there was improvement in the latter audit except for sulfur and moisture parameters. In the 0985, the sulfur and moisture levels were considerably lower, therefore causing a greater chance for error.

TABLE 15. SOURCE COAL AUDIT--0385

Expected value	No. of analyses*	Laboratories accurate within 5% (%)	Laboratories accurate within 10% (%)
		Sulfur	
1.62	(1) 111	76.6	93.7
	(2) 107	78.5	95.3
4.97	(1) 110	53.6	88.2
	(2) 106	60.4	85.8
		Moisture	
3.86	(1) 111	43.2	70.3
	(2) 104	39.4	68.3
3.93	(1) 112	40.2	72.3
	(2) 105	45.7	74.3
		Ash	
11.36	(1) 111	96.4	97.3
	(2) 105	97.1	97.1
20.32	(1) 110	93.6	99.1
	(2) 104	96.2	99.0
		Gross Calorific	
11,414	(1) 108	98.1	99.1
	(2) 103	99.0	99.0
13,054	(1) 109	98.2	99.1
	(2) 104	98.1	99.0

* Numbers in parentheses indicate first and second analyses.

TABLE 16. SOURCE COAL AUDIT--0985

Expected value	No. of analyses	Laboratories accurate within 5% (%)	Laboratories accurate within 10% (%)
		Sulfur	
0.72	(1) 108	46.3	77.8
	(2) 102	50.0	75.5
2.35	(1) 106	62.3	87.7
	(2) 100	65.0	93.0
		Moisture	
1.61	(1) 106	31.1	51.9
	(2) 100	35.0	57.0
17.45	(1) 108	66.7	88.0
	(2) 102	70.6	90.2
		Ash	
4.33	(1) 107	68.2	86.9
	(2) 101	69.3	86.1
17.92	(1) 105	94.3	95.2
	(2) 99	92.9	96.0
		Gross Calorific	
12,300	(1) 102	99.0	100
	(2) 97	100	100
12,374	(1) 104	89.4	93.3
	(2) 99	92.9	92.9

* Numbers in parentheses indicate first and second analyses.

REFERENCES

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2. Streib, E.W., and M.R. Midgett. A Summary of the 1982 EPA National Performance Audit Program on Source Measurements. EPA-600/4-83-049, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina, April 1984.

3. Streib, E.W., T.J. Logan, and M.R. Midgett. A Summary of the 1983 EPA National Performance Audit Program on Source Measurements. EPA-600/4-85-004, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina, January 1985. 38 pp.

4. Streib, E.W., T.J. Logan, and M.R. Midgett. A Summary of the 1984 EPA National Performance Audit Program on Source Measurements. EPA-600/4-86-005, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina, February 1986. 34 pp.

5. Chauvenet, W. Manual of Spherical and Practical Astronomy: Volume II--Theory and Use of Astronomical Instruments (Method of Least Squares). J.P. Lippincott and Co., Philadelphia, Pennsylvania, 1863.

6. American Society for Testing and Materials. Annual Book of ASTM Standards--1984. Volume 5.05. 01-050584-13, American Society for Testing and Materials, Philadelphia, Pennsylvania.

APPENDIX A

FREQUENCY DISTRIBUTIONS

TABLE 17. NATIONAL ORSAT AUDIT FREQUENCY DISTRIBUTION

OF ABSOLUTE PERCENT DIFFERENCES OF EXPECTED AND REPORTED VALUES--STUDY 0785

CO_2

Sample no.	No.	Min.	10%	20%	30%	40%	50%	60%	70%	80%	90%	Max.	Mean	Std.dev.
5000	91	0.00	2.86	2.86	4.29	5.71	5.71	7.14	7.14	10.00	11.43	71.43	8.51	10.85

O_2

Sample no.	No.	Min.	10%	20%	30%	40%	50%	60%	70%	80%	90%	Max.	Mean	Std.dev.
5000	93	0.00	0.00	0.00	0.83	0.83	1.67	1.67	1.67	3.33	4.17	51.67	4.24	10.44

CO

Sample no.	No.	Min.	10%	20%	30%	40%	50%	60%	70%	80%	90%	Max.	Mean	Std.dev.
5000	73	0.00	0.00	0.00	0.00	16.67	16.67	16.67	33.33	50.00	66.67	2950.00	102.06	480.43

TABLE 18. DGM FREQUENCY DISTRIBUTION OF ABSOLUTE PERCENT DIFFERENCE--0685

Sample no.	Min.	10%	20%	30%	40%	50%	60%	70%	80%	90%	Max.	Mean	Std.dev.	Skewness	Median
633	0.0	0.6	1.0	1.3	1.8	2.2	2.6	3.2	4.1	6.3	1,562.9	10.6	102.1	14.34	2.2
630	0.0	0.6	1.0	1.3	1.7	2.2	2.6	3.2	4.1	6.1	75.2	3.6	6.5	-0.00	2.2
617	0.0	0.6	1.0	1.3	1.7	2.2	2.5	3.1	4.0	5.7	20.1	2.8	2.6	-0.00	2.2
609	0.0	0.6	1.0	1.2	1.7	2.1	2.5	3.1	3.9	5.2	11.4	2.6	2.1	-0.00	2.2
600	0.0	0.6	1.0	1.2	1.7	2.1	2.5	3.0	3.7	5.0	9.6	2.5	1.9	-0.00	2.1
591	0.0	0.6	1.0	1.2	1.7	2.0	2.4	2.9	3.6	4.8	8.6	2.4	1.7	-0.01	2.0
588	0.0	0.5	0.9	1.2	1.7	2.0	2.4	2.9	3.6	4.7	7.7	2.3	1.7	-0.01	2.0

TABLE 19. SO_2 FREQUENCY DISTRIBUTION OF PERCENT DIFFERENCE--NO OUTLIERS REMOVED--AUDIT 0585

Sample no.	No.	Min.	10%	20%	30%	40%	50%	60%	70%	80%	90%	Max.	Mean	Std.dev.
2	90	0.00	0.40	0.77	1.08	1.51	1.62	2.15	2.94	4.22	11.05	309.06	8.18	34.50
4	89	0.00	0.35	1.01	2.12	2.67	3.68	4.69	5.65	7.31	15.33	1186.89	26.47	132.35
7	89	0.01	0.35	0.60	0.88	1.12	1.40	1.80	2.55	3.93	6.17	304.61	9.93	44.38
8	91	0.00	0.38	0.73	0.99	1.22	1.66	1.97	2.44	3.17	5.83	296.58	9.38	43.25
9	91	0.00	0.54	0.99	1.81	2.31	3.35	3.48	5.06	6.69	14.69	301.67	13.72	46.52

TABLE 20. NO_x FREQUENCY DISTRIBUTION OF PERCENT DIFFERENCE--NO OUTLIERS REMOVED--AUDIT 0485

Sample no.	No.	Min.	10%	20%	30%	40%	50%	60%	70%	80%	90%	Max.	Mean	Std.dev.
1	73	0.03	0.37	1.51	2.31	3.45	4.39	5.66	7.23	9.21	20.73	79.60	9.23	13.81
3	72	0.10	1.51	2.41	2.61	4.52	6.63	10.05	14.87	26.43	33.97	95.18	15.01	19.43
5	73	0.00	0.65	1.77	2.75	3.48	4.52	6.27	9.08	15.40	32.76	98.81	11.69	17.77
6	72	0.03	0.62	1.63	2.63	3.63	4.43	6.62	9.04	15.48	27.45	117.55	11.61	18.50
8	73	0.00	0.50	1.26	2.09	4.44	4.60	7.95	9.62	19.33	32.13	79.33	11.58	15.31

TABLE 21. NATIONAL COAL AUDIT FREQUENCY DISTRIBUTION
OF ABSOLUTE PERCENT DIFFERENCES OF EXPECTED AND REPORTED VALUES--STUDY 0385

Sulfur

Sample no.	No.	Min.	10%	20%	30%	40%	50%	60%	70%	80%	90%	Max.	Mean	Std.dev.
1000	218	0.00	0.62	1.23	1.85	1.85	3.09	3.70	4.32	5.56	8.02	66.67	4.34	6.98
2000	216	0.00	0.60	1.61	2.21	3.22	4.23	5.23	6.84	7.85	11.47	42.25	5.76	6.22

Moisture

Sample no.	No.	Min.	10%	20%	30%	40%	50%	60%	70%	80%	90%	Max.	Mean	Std.dev.
1000	217	0.00	0.76	1.27	3.05	4.58	6.62	8.14	9.41	11.45	22.39	417.05	13.01	40.70
2000	215	0.00	1.04	2.07	3.37	4.66	6.22	8.03	10.10	15.28	22.54	89.12	10.05	12.30

Ash

Sample no.	No.	Min.	10%	20%	30%	40%	50%	60%	70%	80%	90%	Max.	Mean	Std.dev.
1000	216	0.00	0.09	0.26	0.35	0.44	0.53	0.79	1.06	1.41	2.64	24.12	1.47	3.51
2000	214	0.00	0.25	0.44	0.79	0.94	1.28	1.37	1.87	2.26	3.54	77.36	2.32	7.46

Gross Calorific

Sample no.	No.	Min.	10%	20%	30%	40%	50%	60%	70%	80%	90%	Max.	Mean	Std.dev.
1000	213	0.00	0.08	0.18	0.25	0.36	0.47	0.59	0.78	1.03	1.55	17.59	0.92	1.86
2000	211	0.00	0.09	0.16	0.25	0.33	0.46	0.63	0.77	1.05	1.63	19.98	0.92	2.07

TABLE 22. NATIONAL COAL AUDIT FREQUENCY DISTRIBUTION
OF ABSOLUTE PERCENT DIFFERENCES OF EXPECTED AND REPORTED VALUES--STUDY 0985

Sulfur

Sample no.	No.	Min.	10%	20%	30%	40%	50%	60%	70%	80%	90%	Max.	Mean	Std.dev.
3000	210	0.00	1.39	1.39	2.78	4.17	5.56	6.94	8.33	11.11	18.06	2256.94	31.37	219.04
4000	206	0.00	0.43	0.85	1.70	2.98	3.83	4.26	5.96	8.09	9.79	55.74	5.51	7.60

Moisture

Sample no.	No.	Min.	10%	20%	30%	40%	50%	60%	70%	80%	90%	Max.	Mean	Std.dev.
3000	210	0.11	0.63	1.43	2.12	2.58	3.15	4.01	5.27	6.76	10.09	99.08	6.64	14.28
4000	206	0.00	1.24	2.48	4.35	6.21	8.70	11.18	14.91	19.88	32.30	1009.94	25.77	106.36

Ash

Sample no.	No.	Min.	10%	20%	30%	40%	50%	60%	70%	80%	90%	Max.	Mean	Std.dev.
3000	208	0.00	0.46	1.15	1.62	2.31	3.00	3.70	5.08	6.93	14.09	83.83	5.35	9.10
4000	204	0.00	0.22	0.45	0.61	0.78	1.17	1.45	2.01	2.51	4.13	87.67	3.82	12.53

Gross Calorific

Sample no.	No.	Min.	10%	20%	30%	40%	50%	60%	70%	80%	90%	Max.	Mean	Std.dev.
3000	203	0.00	0.28	0.45	0.61	0.79	0.99	1.12	1.48	1.92	3.51	18.52	2.30	4.19
4000	199	0.00	0.07	0.15	0.24	0.37	0.50	0.64	0.89	1.08	1.63	9.34	0.80	1.04

APPENDIX B

INSTRUCTIONS FOR EPA AUDIT MATERIALS

INSTRUCTIONS FOR USING EPA METHOD 3 AUDIT MATERIALS

Equipment Supplied with Audit Kit

(1) Small gas cylinder containing four liters of gas

(2) Nozzle for cylinder (taped on cylinder)

(3) Tygon tubing

Equipment to be Supplied by Participant

(1) Orsat analyzer

(2) Clamp

(3) Extra Tygon or surgical tubing

Procedure

(1) Leak-check apparatus by clamping off tubing. SEE DIAGRAM.

(2) Fill up burette with gas by using positive pressure. Vent this sample through the manifold to the atmosphere.

(3) Repeat Step 2.

(4) Fill up burette past fill mark with the gas and carefully vent out excess to the atmosphere, until the fill mark is reached.

(5) Analyze for CO_2, O_2 and CO as described in Sections 4.2.5, 4.2.6 and 4.2.7 of EPA Method 3.

(6) Record the results on the data card enclosed with the sample.

(7) Repeat Steps 4 through 6.

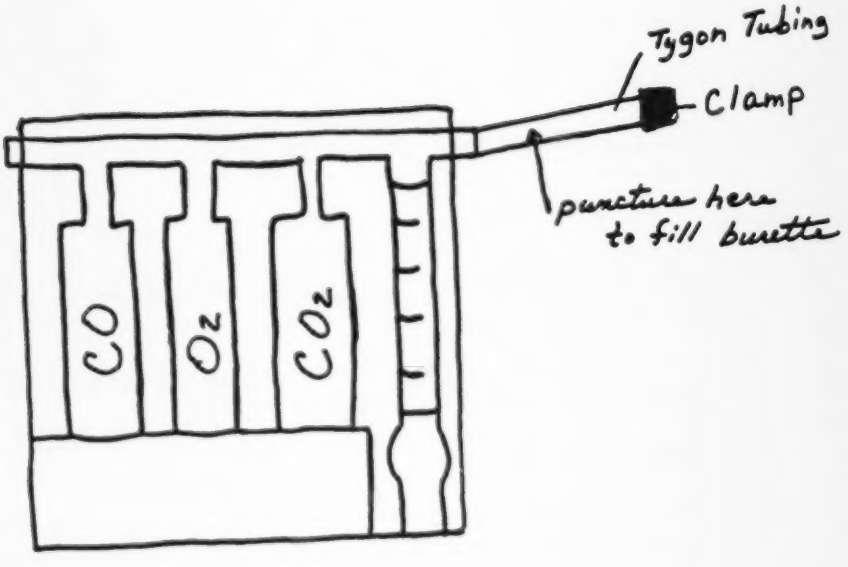
CAUTION: If the tubing is punctured excess times, leakage can occur. Replace if necessary.

Send the data card to the address below. (The cylinder gas can should not be returned.)

Ms. Ellen Streib
Quality Assurance Division (MD-77A)
Environmental Monitoring Systems Laboratory
U.S. Environmental Protection Agency
Research Triangle Park, NC 27711

Note: Site number will always be 001 except when other Orsat apparatus or participants are using the same gas sample. The extra apparatus or participants should be labeled 002, 003, etc.

APPARATUS SET-UP



INSTRUCTIONS FOR USE OF ENVIRONMENTAL PROTECTION AGENCY METHOD 5 DRY GAS METER PERFORMANCE TEST DEVICE

NOTE: All procedures referred to are from revised Method 5 published in the Federal Register, Volume 12, Number 160, Part II, Thursday, August 18, 1977, pp. 41776-41782 and references contained therein. This revised method should be adhered to in all details in the use of this quality assurance performance device.

EQUIPMENT: The participant in this study should possess the following equipment, including the performance test device supplied by EPA.

Quantity	Item
1	Method 5/Source Sampling Meter Box
1	Stopwatch, preferably calibrated in decimal minutes
1	Thermometer, ambient range
1	Barometer. If unavailable, call nearest National Weather Service and request the ABSOLUTE barometric pressure. (Corrected for temperature and acceleration due to gravity, but not corrected for altitude.)
1	Performance Test Device. A calibrated flow orifice housed in a quick-connect coupling and identified with an engraved three-digit serial number. WARNING: THE DEVICE MUST NOT BE DISASSEMBLED UNDER ANY CIRCUMSTANCES. Use these devices at room temperature.

PROCEDURE:

1. Calibration of Vacuum Gauge -- The vacuum pressure gauge on the meter box must be calibrated in the range of use (11-22" Hg) against a standard (Hg Manometer) to ensure accurate results.

2. Remove the performance test device from its case and insert it into the gas inlet quick-connect coupling on the source sampling meter box.

3. Turn the power to the meter box on and start the pump.

4. Adjust the coarse flow rate control valve and the fine flow rate control valve to give a reading of 19" Hg (vacuum reading). CAUTION: The vacuum reading must be accurate and stable for the test period.

5. Allow the orifice and source sampling meter box to warm up for 45 minutes with flow controls adjusted as described in Step 3 before starting quality assurance runs.

PROCEDURE: (continued)

6 Make triplicate quality assurance runs. For each run, record initial and final dry gas meter volumes, dry gas meter inlet and outlet temperatures, internal orifice pressure drop (ΔH), ambient temperature, and barometric pressure. Run duration should be slightly greater than 15 minutes. The following procedure is recommended. Fifteen minutes after a run is started, the participant watches the dry gas meter needle closely. As the needle reaches the zero (12 o'clock) position, the pump and stopwatch are stopped simultaneously. The dry gas meter volume and time are recorded.

This complete run procedure is performed three times to provide the required triplicate quality assurance runs.

7 Calculate the corrected dry gas volume for each run using equation 5.1 of the above-referenced Method 5. For each replicate, record the corrected dry gas volume in dry standard cubic meters, the sampling time in decimal minutes, the barometric pressure in mm Hg, and the ambient temperature in degrees Celcius on the enclosed data card. Be sure to record the performance test device serial number on the data card in the column headed "Orifice Number."

NOTE 1: If you calculate dry gas volume in English Units, use the conversion factor of 0.02832 m^3ft^3 to obtain the volume in metric units.

NOTE 2: If your stopwatch is not in decimal minutes, be sure to convert (e.g. 15 minutes 20 seconds is reported as 15.33 minutes).

8 After recording the requested data on the enclosed data form, return the data form and the performance test device to:

Quality Assurance Division (MD-77A)
Environmental Monitoring Systems Laboratory
U.S. Environmental Protection Agency
Research Triangle Park, NC 27711
ATTN: Ellen W. Streib

A postpaid return envelope and label are enclosed for this purpose.

INSTRUCTIONS FOR USE OF ENVIRONMENTAL PROTECTION AGENCY STATIONARY SOURCE QUALITY ASSURANCE SO_2 REFERENCE SAMPLES

NOTE: All Method 6 procedures referred to are from the amended method published in the Federal Register Vol. 42, No. 160, Part II, Thursday, August 18, 1977, pp 41782-41784. This amended method should be adhered to in all details in the analysis of these reference standards.

1. Prepare 3 percent hydrogen peroxide according to Section 3.1.3 of the method (30 ml is required for each sample and each blank).

2. Prepare each reference sample for analysis as follows: Wrap a paper towel around the ampoule and with the ampoule in an upright position break off the top at the prescored mark by exerting pressure sideways. From the ampoule pipette exactly 5 ml of the reference sample into a 100 ml volumetric flask. Add 30 ml of 3 percent hydrogen peroxide solution. Dilute exactly to the mark with deionized, distilled water. Analyze the sample in accordance with the procedure detailed in Section 4.3 of the method, beginning with "Pipette a 20 ml aliquot of this solution..." (Note: If more than 50 ml of barium perchlorate titrant is required for any sample analysis, a smaller aliquot should be selected to allow titration with less than 50 ml titrant.)

3. Calculate the concentration, C_{SO2} (concentration of sulfur dioxide, dry basis, corrected to standard conditions, mg/dscm), using Equation 6-2. A value of 21×10^{-3} dscm should be used for $V_m(std)$, in the equation. A value of 100 ml should be used for V_{soln} in the equation.

4. Record the reference standard sample numbers and their corresponding SO_2 concentrations in mg/dscm on the enclosed data form. Return the form to:

Quality Assurance Division (MD-77A)
Environmental Monitoring Systems Laboratory
U.S. Environmental Protection Agency
Research Triangle Park, NC 27711
ATTN: Ellen W. Streib

If other than EPA Method 6 is used for your analyses, please explain in detail your analytical procedure on the back of the enclosed data form.

INSTRUCTIONS FOR USE OF ENVIRONMENTAL PROTECTION AGENCY STATIONARY SOURCE QUALITY ASSURANCE NO_x REFERENCE SAMPLES

Note: All Method 7 procedures referred to are from the amended method published in the Federal Register Vol. 42, No. 160, Part 11, Thursday, August 18, 1977, pp 41784-41786. This amended method should be adhered to in all details in the analysis of these reference standards.

1. Prepare absorbing solution according to Section 3.1 of the method.

2. Prepare each reference sample for analysis as follows: Wrap a paper towel around the ampule and with the ampule in an upright position break off the top at the prescored mark by exerting pressure sideways. From the ampule pipette exactly 5 ml of the reference sample into a 100-ml beaker. Add 25 ml absorbing solution to the beaker; adjust the pH to 9-12 (using pH paper as indicated in Section 4.2 of the method) by dropwise addition of sodium hydroxide (1N). Quantitatively transfer the contents of the beaker to a 50-ml volumetric flask and dilute exactly to the mark with deionized, distilled water. Mix thoroughly and pipette a 25-ml aliquot of the diluted sample into a porcelain evaporating dish. Beginning with the evaporation step in Section 4.3, complete the sample analysis.

3. Calculate total µg NO_2 per sample using Equation 7-3. Calculate the sample concentration, C (concentration of NO_x as NO_2, dry basis, corrected to standard conditions, mg/dscm), using Equation 7-4. A value of 2000 ml should be used for V_{sc} in Equation 7-4.

4. Record the reference sample numbers and their corresponding concentrations, C, in mg/dscm on the enclosed data form. Return the form to:

Quality Assurance Division (MD-77A)
Environmental Monitoring Systems Laboratory
Environmental Protection Agency
Research Triangle Park, NC 27711
ATTN: Ellen W. Streib

If other than EPA Method 7 is used for your analyses, please explain in detail your analytical procedure on the back of the enclosed data form.

COAL AUDIT PROGRAM INFORMATION

1. There is approximately 50 grams of 60 mesh coal per bottle.

2. Analyze the coal samples for moisture and on a dry basis for ash, sulfur and gross calorific value. Report moisture, ash, and sulfur in weight percent with gross calorific value reported as BTU/lb.

3. All methods used in the analysis of these coal samples should follow American Society for Testing and Materials (ASTM) recommended procedures or an accepted automatic analytical device.

4. Suggested procedures are:

Moisture	D-3173
Ash	D-3174
Sulfur	D-3177
Gross Calorific Value	D-2015

   Please note on the data card (columns 17-32) the ASTM method number. If an ASTM method was not used for analysis note that on the back of the data card. Be parameter specific.

5. If you cannot analyze the coal sample for all four parameters, analyze for what you can. Analysis of moisture is necessary to calculate on a dry basis any of the other three parameters. Analysis of sulfur is also necessary for the calculation of gross calorific value.

6. Analyze each sample in duplicate (if possible) and record results as analysis 1 and analysis 2 for each parameter.

7. Most laboratories will use site number 001. Multiple site numbers are used by laboratories that receive more than one set of samples. These central laboratories have requested auditing of their satellite laboratories.

8. After recording the requested data on the enclosed data card, return the data card to:

   Ms. Ellen W. Streib
   Quality Assurance Division (MD-77)
   Environmental Monitoring Systems Laboratory
   U.S. Environmental Protection Agency
   Research Triangle Park, NC 27711

   A postpaid return envelope is enclosed for this purpose.

9. If you have any questions concerning this or any source method audit, please call (919/541-7834).

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